AD-A031 018 BATTELLE COLUMBUS LABS OHIO F/G 11/6

GRAIN ASPECT RATIO STRENGTHENING OF DISPERSION HARDENED METALS. (U)

1976 A H CLAUER, B A WILCOX AF-AFOSR-2083-71

UNCLASSIFIED AFOSR-TR-76-1086 NL

1 OF 1

ADA031018

END

DATE

FILMED

11 - 76

Unclassified
SECURITY CLASSIFICATION O

19 REPORT

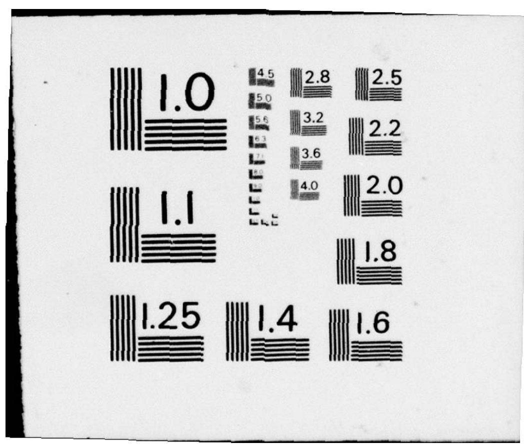
1. REPORT NUMBER
18 AFOSR - TR - 7

4. TITLE (and Subtitle)
6 Grain Aspect Rat
Hardened Metals

7. AUTHOR(s)
10 A. H. Clauer an

9. PERFORMING ORGANIZAT
Battelle-Columb
505 King Avenue
Columbus, Ohio

11. CONTROLLING OFFICE


1.0
4.5
5.0
5.6
6.3
2.8
3.2
3.6
4.0
2.5
2.2
2.0
1.1
1.8
1.25
1.4
1.6

AD A031018

FG.

AFOSR - TR - 76 - 1086

FINAL REPORT

Sle 1473

on

GRAIN ASPECT RATIO STRENGTHENING OF DISPERSION HARDENED METALS

12

Grant No. AFOSR-71-2083

to

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

Covering the Period

May 1, 1971 to April 31, 1976

by


A. H. Clauer and B. A. Wilcox





BATTELLE
Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201


Approved for public release;
distribution unlimited.

FINAL REPORT

on

# GRAIN ASPECT RATIO STRENGTHENING OF DISPERSION HARDENED METALS

to

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH

Covering the Period
May 1, 1971 to April 31, 1976

by

A. H. Clauer and B. A. Wilcox

from

BATTELLE
Columbus Laboratories



## ABSTRACT

This program consisted of investigations into the influence of microstructure on the high temperature creep strength of materials. Three separate studies were undertaken. The first study, "High Temperature Creep of Ceramic Oxides", was directed at the effect of a pre-existing substructure on the creep and tensile behavior of magnesium oxide (MgO) single crystals. In this program, the initial dislocation substructure was modified before creep such that the substructure consisted of either well-defined subgrains or a uniform dislocation distribution. This was done by pre-straining the specimens in tension at 1800 C. Subsequent creep at 1400 C showed no influence of either type of substructure in the early stage of creep compared to the annealed specimens containing no substructure which were studied in a previous program. However, in the "steady state" region, the creep rates were higher in the prestrained specimens, and those specimens containing a uniform dislocation distribution had higher creep rates than those containing subgrains. Thus, the additional dislocation substructure produced by prestraining introduced a weakening effect rather than the

sought-after strengthening. The conclusion was that a heavy dislocation substructure does not automatically enhance high-temperature creep strength. Under conditions where the substructure is not adequately pinned either by precipitates, solutes, or elements of the substructure itself, the effect of prestrain is to raise the mobile dislocation density and thereby raise the creep rate.

The second study "Dislocation Substructure Strengthening in a Dispersion Hardened Alloy", was an investigation of the efficiency of dislocation substructure strengthening in an oxide-dispersion strengthened Ni-20 wt% Cr-2 vol% $ThO_2$ alloy (TD-NiCr). As-received TD-NiCr was prestrained in tension at room temperature to 2, 4 and 6% strain, then annealed at 950 C. The specimens were then compared in creep at 871 C. The dislocation density in the prestrained specimens was raised to 3 to 5 times the density in the as-received material. It was found that the 2% prestrain did impart a modest strengthening increment, as measured by the minimum creep rate, but the 4 and 6% prestrains did not. Although the dislocation substructure did provide some strengthening in addition to that due to the oxide dispersion, the effect was small and appeared to be sensitive to the amount of prestrain introduced. It was concluded that substructure strengthening is not very effective in dispersion hardened alloys.

The third study, "Grain Aspect Ratio Strengthening of Dispersion Hardened Metals", was an investigation of the temperature and strain rate dependence of the grain aspect ratio effect in an oxide-dispersion-strengthened nickel alloy (Ni-2 vol% $ThO_2$). The grain aspect ratio (G.A.R.) is the length to width ratio of the grains. The grain aspect ratio is important under conditions where grain boundary sliding is an important creep mechanism, as it is in dispersion hardened metals having hard grains. Stressing parallel to the long direction of the grains decreases the average shear stress on the grain boundaries and thereby decreases grain boundary sliding. As the G.A.R. increases, the creep and tensile strength increase to a maximum value equal to the Orowan stress, which is the strength of the grain interiors. Specimens having different G.A.R.'s were tensile and creep tested at 1093, 871 and 600 C. It was found that at 871 and 1093 C, the strength increase with increasing G.A.R. was due to inhibition of grain

boundary sliding, but at 600 C the strengthening resulted from the combined effect of the inhibition of grain boundary sliding and grain refinement (Hall-Petch strengthening). The G.A.R. effect could be reproduced by combining creep equations for the bulk creep behavior and grain boundary sliding. In addition, creep deformation maps were derived from TD-Ni. Creep studies on coarse-grained Ni-20wt%Cr-2vol% $ThO_2$ alloy had to be discontinued because of the inhomogeneous grain microstructure in the material.

## SUMMARY OF RESULTS

This program has consisted of three studies that will be discussed under separate headings.

### High Temperature Creep of Ceramic Oxides

This study had the objective of investigating the influence of different types of dislocation substructure on the tensile creep behavior of MgO single crystals. A previous study of creep of annealed single crystals had shown that two types of dislocation substructure formed during tensile creep at 1400 C. At low stresses blocky subgrains formed, while at higher stresses a uniform distribution of dislocations developed.

This behavior provided the opportunity to investigate the recurring question of the effect of dislocation substructures on high temperature creep strength. Single crystal specimens were prestrained to 10% tensile strain at 1800 C at different strain rates to produce a high density of dislocations both in a uniform distribution and in blocky subgrains. These specimens were then creep tested at 1400 C and their behavior and substructural changes compared with those of the annealed specimens.

The prestrained specimens behaved identically to the unstrained specimens in the early stages of creep. However, in the "steady state" region the prestrained specimens had higher creep rates, e.g., lower creep strength. Also, the specimens having a uniform distribution of dislocations had higher creep rates than those containing the subgrains. During creep

the different initial microstructures evolved in different ways. The initially homogeneous substructure remained that way while the initially blocky subgrain substructure changed to elongated subgrains.

The reason for this difference is probably as follows. On the one hand the uniform dislocation substructure is composed of dislocations from predominantly one <110> $\{1\bar{1}0\}$ slip system in which there are no interactions which result in sessile product dislocations to anchor and nucleate subgrain boundaries. On the other hand, the blocky subgrain structure is formed by the interaction of dislocations from oblique <110> $\{1\bar{1}0\}$ slip systems which form sessile product dislocations. These sessile dislocations provide the basis for the subgrain boundary formation during the prestraining at 1800 C. During subsequent creep at 1400 C under conditions where no subgrains form in annealed specimens, no further sessile dislocations are formed and those that are already present rearrange themselves by climb, and possibly by continuing interactions, to develop long, unconnected subgrain boundaries.

Thus a pre-existing dislocation substructure does not automatically impart creep strength. Unless it is strongly pinned by strong solute interactions or a finely dispersed second phase, the dislocations move under the applied creep stress and provide a large number of mobile dislocations compared with the annealed specimens. In this case the creep rates are higher when a dislocation substructure is present. The partially pinned substructure containing subboundaries anchored by sessile dislocations was slightly stronger than the subgrain-free substructure because the boundaries provided barriers to dislocation glide which required some additional stress to overcome.

## Dislocation Substructure Strengthening in TD-NiCr

Because the above results showed that a strongly pinned dislocation substructure is required to impart high-temperature substructural strengthening, an investigation of dislocation substructure strengthening in the presence of a finely dispersed second phase was undertaken. Also, the role of the dislocation substructure in the strengthening dispersion hardened

alloys was in question. The authors had originally attributed some of the high-temperature strength of TD-Ni to a fine subgrain structure.

Tensile specimens machined from 0.050 inch-thick sheet of TD-NiCr (nickel-20wt% chromium -2 vol.% $ThO_2$) were prestrained in tension to 2, 4 and 6% strain at room temperature, then annealed one hour at 950 C to stabilize the substructure. The prestrained and annealed specimens had dislocation densities of 7 to 10 x $10^9$ $cm^{-2}$, about 3 to 5 times the dislocation density of the unstrained specimens.

The creep behavior was compared at 871°C over a range of creep stress. The specimens given the 2% prestrain had a lower minimum creep rate than the unstrained specimens at all except the highest stresses, whereas the 4 and 6% prestrained specimens showed no significant difference from the unstrained material. However, even the 2% prestrain imparted only a modest increase in creep strength.

These results seem to indicate either of two effects. Firstly, a dislocation substructure can provide additional strengthening above that provided by the oxide dispersion as shown by the 2% prestrain results. It would be expected that this increment would increase with increasing prestrain (increasing dislocation density) since the total strength is some monotonically increasing combination of the flow stress dictated by the oxide particles and the dislocations. That this was not observed suggests that there is possibly a sensitive relation between the characteristic length of the dislocation network and the oxide dispersion. If these conditions are not met, then during creep at high temperatures sufficient rearrangement and annihilation occurs within the dislocation substructure by climb and glide, that any benefit is soon dissipated. Secondly, it is possible that the tensile prestrain at room temperature introduces an incipient neck within the gage section. During the subsequent creep test premature failure will occur in this necked region compared with the unstrained or 2% prestrained specimens. This would have the result that apparently higher minimum creep rates would be measured which would offset a small strengthening increment. If premature failure did occur, then lower total elongations should be observed in the 4 and 6% prestrained specimens. However, the total elongations to failure for all the specimens were similar.

It was concluded that in either case, any additional strengthening contributed by a dislocation substructure in a dispersion hardened alloy would be small compared to the dispersion strengthening contribution. The stable mesh size of any dislocation network would rapidly change to that of the dispersion spacing or greater during creep at high temperatures. Thus since the strengthening contribution increases with the obstacle spacing, the dislocation network becomes a less effective strengthener at high temperatures compared to that of the dispersion.

## Grain Aspect Ratio Strengthening of Dispersion Hardened Metals

Dispersion hardened metals often have microstructures consisting of elongated grains with the grains elongated in the primary working direction. These materials are often tested and used such that the principal stress is parallel to the long dimension of the grains. It was discovered by the authors several years ago that the high-temperature strength increased with increasing elongation of the grains until a plateau of mixmum strength was reached and thereafter no further strengthening accrued from increased elongation of the grains. The degree of elongation was measured as the ratio of the length to the diameter of the grains and was called the grain aspect ratio (G.A.R.).

This effect occurred because the grains were substantially hardened by the oxide particles, leaving the grain boundaries as the weak link in the microstructure and causing grain boundary sliding to be an important creep mechanism in these materials. Increasing the G.A.R. decreased the average shear stress on the grain boundaries and decreased grain boundary sliding when the material was stressed parallel to the long dimension of the grains. Thus the strength increased with increasing G.A.R. until the strength of the grains was reached at the Orowan stress, and after this point further grain elongation provided no increase in strength.

These results had been obtained on TD-Ni (Ni-2 vol % $ThO_2$) at a temperature (1093 C) well above half the melting temperature. It was desirable to know whether this effect persisted to lower temperatures and a wide range of strain rates, both for practical and scientific reasons.

In this program, TD-Ni rod was reduced by swaging to decreasing diameters with the net effect of elongating the grains and increasing the G.A.R. These specimens were tested in tension and tensile creep at 871 and 600 C over a wide range of strain rates from 1 $min^{-1}$ to $1.7 \times 10^{-7}$ $min^{-1}$. Some of the drawn material was given an intermediate anneal of 1 hr at 1200 C between cold reductions while the rest of the material had no intermediate anneals. A range of G.A.R. from 1.7 to 31 was obtained by this method and these grains were stable at both 600 and 871 C.

The stress dependence of the minimum creep rate was very high and tended to increase slightly with increasing G.A.R. up to n = 90 for the relation $\dot{\varepsilon} \propto \sigma^n$. At constant temperature and strain rate the creep and yield strength increased linearly with G.A.R. up to a G.A.R. of 10 to 15. Above G.A.R. ≅ 15 the strength was either constant or increased slowly with G.A.R. The initial linear increase of strength, $\sigma$, with G.A.R. can be described by the relation

$$\sigma = \sigma_e + K \left(\frac{L}{\ell} - 1\right) \quad (1)$$

where $\sigma_e$ is the strength of material having equiaxed grains, L is the grain length, $\ell$ is the grain width, and K is defined as the grain aspect ratio coefficient. The ratio $L/\ell$ is called the G.A.R.

At 871 C, K was independent of strain rate for rates in the range of $10^{-7}$ to 1 $min^{-1}$ and was 8.7 $MN/m^2$. The major effect of increasing strain rate was to shift the curves to higher strength levels, but there was also a tendency for the strength to increase with increasing G.A.R. in the plateau region. This is probably due to the continued refinement of grain diameter with increasing G.A.R. in this material, giving rise to a small Hall-Petch strengthening contribution. In a previous study, the room temperature strength followed the Hall-Petch relation very well.

At 600 C, K was larger, up to 17.9 to 26.2 $MN/m^2$ and was not as reproducible as at 871 C. The strength plateau was reached in the range of G.A.R. = 10 to 15 independent of strain rate at 871 C, but there was much more dependence of strength on the G.A.R. in the plateau region at 600 C. This is because the Hall-Petch strengthening contribution becomes more important with decreasing temperature.

Summarizing, a comparison of the strength variation at 600, 871 and 1093 C at constant strain rate clearly shows that K is constant with decreasing temperature down to 871 C and then increases at lower temperatures. The end of the linear variation of stress with temperature is independent of temperature and strain rate. The strength variation in the plateau region above G.A.R. = 15 is nearly independent of G.A.R. at low strain rates and high temperatures, but the strength begins to increase with increasing G.A.R. at lower temperatures and higher strain rates due to the increasing importance of Hall-Petch strengthening.

The relative independence of strength with increasing G.A.R. > 15 is caused by the inhibition of grain boundary sliding and predominance of deformation of the grains themselves. The strength is controlled by the strength of the grains and this is related to the Orowan stress, the stress required to bow dislocations past the oxide particles within the grains. At the higher temperatures this is the only strengthening mechanism and the strength is independent of G.A.R. The calculated Orowan stresses at 871 C and 1093 C agree very well with the observed strengths of the plateau region at these temperatures, but the strength of the plateau region at 600 C is substantially higher than the calculated Orowan stress. This difference at 600 C is the result of the increasing importance of the low temperature strengthening mechanisms related to grain refinement and dislocation substructure.

The variation of high temperature strength with increasing G.A.R. was described by combining expressions for grain boundary sliding (R. Raj and M. F. Ashby, Met. Trans., 2, 1113 (1971)) and power law creep in oxide dispersion hardened alloys. The combined form is

$$\dot{\epsilon} = \frac{A\ D_v \sigma}{d^2 (\frac{L}{\ell})^2} \ (1 + \frac{B(\frac{L}{\ell})\ D_B}{D_v}) + C\ (\sigma - \sigma_o)^n \qquad (2)$$

where A, B, C and n are constants, $D_v$ and $D_B$ are the volume and grain boundary diffusion coefficients respectively, d is the grain diameter, $\frac{L}{\ell}$ is the grain aspect ratio, $\dot{\epsilon}$ is the strain rate, $\sigma$ is the applied stress and $\sigma_o$ is the Orowan stress. The flow stress is calculated by inserting the appropriate physical and empirical values for the different parameters. A

computer iteration procedure is required for the solution which then gives values for $\sigma$ with increasing $\frac{L}{\ell}$. This approach has given the appropriate form of the curve, i.e., increasing strength with increasing $\frac{L}{\ell}$ up to a constant strength equal to the Orowan stress. However, in the calculated form the transition value of G.A.R. at which the constant flow stress occurs is temperature dependent and decreases with decreasing temperature, becoming lower than the experimental value of G.A.R. ≈ 15.

In a study of coarse grained TD-NiCr it was shown that coarse grained material with long grain aspect ratio had a considerably higher stress dependence and was considerably stronger than the material having equiaxed grains. However, the study of the influence of grain aspect ratio in coarse-grained TD-NiCr was discontinued after it was ascertained that the microstructures were too heterogeneous and the wide variation in grain size and aspect ratios within the specimens was too large to give data amenable to analysis.

## COUPLING

Grant No. AFOSR-71-2083 Date: September 1, 1976

1. Influence of Grain Aspect Ratio on High Temperature Strength
    a. A. H. Clauer
    b. Seminar
    c. A seminar was presented at the University of Connecticut based on this program. The importance of an increasing grain aspect ratio and the existence of a threshold creep strength equal to the Orowan stress was discussed.

2. Strength of Oxide Dispersion Hardened Metals
    a. A. H. Clauer
    b. Discussion
    c. A discussion of dispersion strengthening was held with Dr. N. Hansen of the Danish Atomic Energy Commission Risø Laboratories. They are pursuing work in oxide dispersion strengthened stainless steels and aluminum. The importance

of the grain aspect ratio and a threshold stress in terms of thermomechanical processing to achieve the appropriate microstructure was discussed.

3. Strength of Oxide Dispersion Strengthened Metals
   a. A. H. Clauer
   b. Discussion
   c. A discussion of strengthening in nickel base oxide dispersion hardened alloys was carried out with Dr. V. S. Arunachalam, Director of the Indian Defense Metallurgical Research Laboratory. Possible mechanisms for by pass of the oxide particles and their relation to a high temperature threshold stress was discussed.

4. Dispersion Strengthening on Ductilizing of TiAl,
   a. A. H. Clauer, B. A. Wilcox and I. G. Wright
   b. Project
   c. Discussions were held with Dr. H. Lipsitt of the Air Force Materials Laboratory concerning the possibility of dispersion strengthening or ductilizing TiAl using a fine oxide dispersion of 1.5 to 3 vol.% $Y_2O_3$. This was subsequently investigated in a small program. Unfortunately the dispersion hardening was nominal and the ductilizing non-existent.

## PUBLICATIONS

### Papers in Preparation

1. "The Role of Dislocation Substructure in Creep of MgO Single Crystals", by A. H. Clauer, M. S. Seltzer and B. A. Wilcox.

2. "Dislocation Substructure Strengthening in TD-Ni-Cr", by A. H. Clauer and B. A. Wilcox.

3. "Computer Simulation of a Dislocation Network", A. H. Clauer.

4. "The Temperature and Strain Rate Dependence of the Grain Aspect Ratio Effect", A. H. Clauer, B. A. Wilcox, and A. J. Markworth.

## PERSONNEL

The scientific personnel involved in this program have been Dr. A. H. Clauer, Dr. B. A. Wilcox, Dr. M. S. Seltzer and Dr. A. J. Markworth.

Unclassified
SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

# 19 REPORT DOCUMENTATION PAGE

READ INSTRUCTIO BEFORE COMPLETING

1. REPORT NUMBER
18 AFOSR - TR - 76 - 1086

2. GOVT ACCESSION NO.

3. RECIPIENT'S CATALOG NUM

4. TITLE (and Subtitle)
6 Grain Aspect Ratio Strengthening of Dispersion Hardened Metals.

5. TYPE OF REPORT & PERIOD
9 Final Report. 1 - 31 Apr 76

6. PERFORMING ORG. REPORT

7. AUTHOR(s)
10 A. H. Clauer ~~and~~ B. A. Wilcox

8. CONTRACT OR GRANT NUMB
15 AF-AFOSR-2083-71

9. PERFORMING ORGANIZATION NAME AND ADDRESS
Battelle-Columbus Laboratories
505 King Avenue
Columbus, Ohio 43201

10. PROGRAM ELEMENT, PROJE AREA & WORK UNIT NUMBE

11. CONTROLLING OFFICE NAME AND ADDRESS
AF Office of Scientific Research (NE)
Bolling Air Force Base, Building 410
Washington, D. C. 20332

12. REPORT DATE

13. NUMBER OF PAGES

14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)
11 1976 12 14p.

15. SECURITY CLASS. (of this re
Unclassified

15a. DECLASSIFICATION/DOWN SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)
nickel alloys, oxide dispersion strengthening, creep of Ni-$ThO_2$ allo creep, grain aspect ratio, grain shape effect on creep, magnesium ox dislocation substrcture, strengthening

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

The effect of a prestrain dislocation substructure on the tensi of MgO single crystal specimens compared with annealed specimens was investigated. Specimens having a substructure consisting of subgrai slightly stronger than those having a uniform dislocation distributi the strongest condition were annealed specimens having few dislocati Without strong pinning, the prestrain dislocation substructure merel provided a higher density of mobile dislocations. Substructural st

DD FORM 1473 1 JAN 73 EDITION OF 1 NOV 65 IS OBSOLETE

Unclassified
SECURITY CLASSIFICATION OF THIS PAGE (

40

ing of an oxide dispersion hardened metal, TD-NiCr showed that a dislocation substructure added only a modest amount to the creep strength and the effect appeared to be sensitive to the level of prestrain used. The temperature and strain rate dependence of the grain aspect ratio effect on TD-Ni was investigated at 871 and 600 C over a range of strain rate. The strengthening effect of elongated grains persisted down to 600°C although strengthening due to grain refinement also contributed at 600°C. The strength increased with increasing grain aspect ratio (G.A.R.) linearly up to a ratio of 10 to 15, after which the strength increased only slowly with further increase in G.A.R. This limit of G.A.R. = 10 to 15 was independent of temperature and strain rate. An expression combining grain boundary sliding and deformation of the grains duplicated the form of the G.A.R. effect on creep strength.